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Several new experimental techniques were used for a comprehensive study of the microstructures and physical properties of high transition-temperature superconductors. This research made extensive use of synchrotron radiation which allowed many unique ways to probenon-destructively the short-range-order structure in superconductors. In addition, some novel methods were used to investigate the effects of chemical doping and transport as well as magnetic properties of thin films of superconducting materials prepared by laser ablation.

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FINAL TECHNICAL REPORT

Grant Number: AFOSR-88-0095

Title: X-ray Absorption Studies of High Transition Temperature Superconductors

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Summary:

Several new experimental techniques were used for a comprehensive study of the microstructures and physical properties of high transition-temperature superconductors. This research made extensive use of synchrotron radiation which allowed many unique ways to probe nondestructively the short-range-order structure in superconductors. The synchrotron experiments were performed at the National Synchrotron Light Source in Brookhaven National Laboratory. In addition, some novel methods were used to investigate the effects of chemical doping and transport as well as magnetic properties of thin films of superconducting materials prepared by laser ablation at Buffalo.

I. INTRODUCTION

This is the final report of the grant AFOSR-88-0095 covering the period from January 1, 1988 to July 31, 1991.

An outstanding property of the oxide superconductors is that the coherence length ($\xi \sim 10$Å) is much smaller than that of conventional low-T_c superconductors. This implies that real space pairing of electrons plays an important role, and Cooper pairs can be bound by a non-retarded potential. Moreover, the Fermi energy is comparable to the binding energy, pairs could exist at temperatures above T_c, and dynamic charge fluctuations in real space should be closely related to the superconducting properties of the material. It is plausible that changes in the local microstructure within ξ of the material, e.g. near an interface or in the vicinity of impurity atoms by chemical doping, could allow an effective means to control the superconducting properties. A complete explanation of the high-T_c phenomena, as well as many device applications of these superconductors, can only be achieved with a clear understanding of the dependence of local bonding and electronic structure on the scale of ξ. To this end, we initiated a program to investigate the short-range structural order and to develop effective characterization methods for exploring the microstructures in the high-T_c superconductors.

Special efforts were also devoted to a better understanding as well as detailed control of weak links, and pinning, creep, motion of the magnetic flux lines. Several experiments were designed to study their effects on critical current density and high-current-carrying capacity and new phenomena were discovered. Of great interest is the correlation between the effects of chemical doping and changes in critical current density.

It was really unfortunate that AFOSR decided to discontinue supporting our active research on these important problems, just at the peak of our high productivity.

II. SCIENTIFIC RESULTS

The experimental methods used in this research include some features which are uniquely suited for the study of chemical-doping and thin films of high-T_c oxide superconductors. Our experiments made effective use of synchrotron radiation to probe the short-range-order structure in oxide superconductors based on our unique capability and extensive experience. These experiments were performed at National Synchrotron Light Source (NSLS). Various magnetic and transport properties were studied in our new laboratories at Buffalo. Laser ablation and laser patterning techniques were employed to prepare specific material systems for our experimental investigation. We are using many state-of-the-art techniques for these measurements, including some experimental methods first used by our group.

(II-A). Material Studies with Synchrotron Radiation

The advent of high intensity polarized x-rays from synchrotron radiation provides unique opportunities to probe microscopic structures in multi-component material systems in ways which were not possible before. Our group has been actively using several different x-ray techniques including fluorescence, grazing angle reflectivity, electron yields, and x-ray absorption fine structure (XAFS) for extensive studies of various multi-element and multi-

layer materials. As our results have already demonstrated, these techniques are very useful and well suited for probing the local environment around specific atomic species, impurity atoms, interfaces, and local electronic structure on selected atomic sites. These methods are particularly useful for our investigation of the oxide superconductors and chemical doping.

Of special interest is our success in the development of a new detector for measuring the fluorescence yield from low-Z elements. This has proved to be particularly useful to study the local environment and electronic structure about the oxygen atoms in high-Tc superconductors. Moreover, the energy and angular variation of oxygen fluorescence can also be employed as a tool for *nondestructive characterization* of microstructures in these superconductors. This study of oxygen is especially significant in light of the fact that oxygen is the only common element found in all the high-T_c superconductors known to date, and the important charge carriers (holes) are largely located around the oxygen sites.

Our experiments are routinely conducted at two beamlines (X3 and U15) at NSLS to cover both the hard and soft x-ray regions. Some measurements were also performed at NSLS Beamline U4, and at Cornell High Energy Synchrotron Source (CHESS) to make use of some special x-ray characteristics at these stations.

X-ray Absorption Fine Structure (XAFS) Spectroscopy

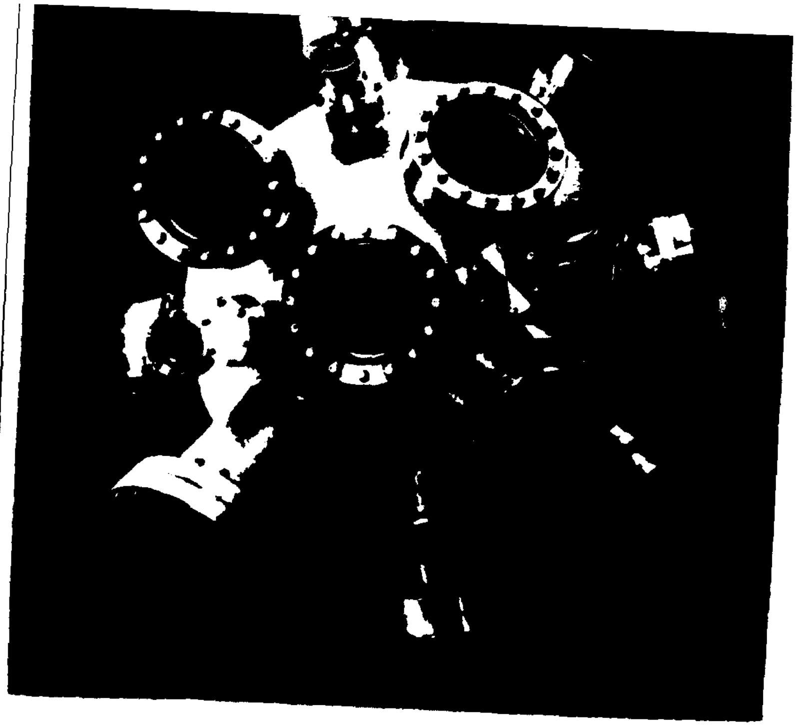
XAFS generally refers to extended x-ray absorption fine structure (EXAFS) and near edge x-ray absorption fine structure (NEXAFS or XANES), these spectra contain valuable information on the local environment and electronic structure about selected atoms in materials. Detailed description of these techniques can be found in several review articles.

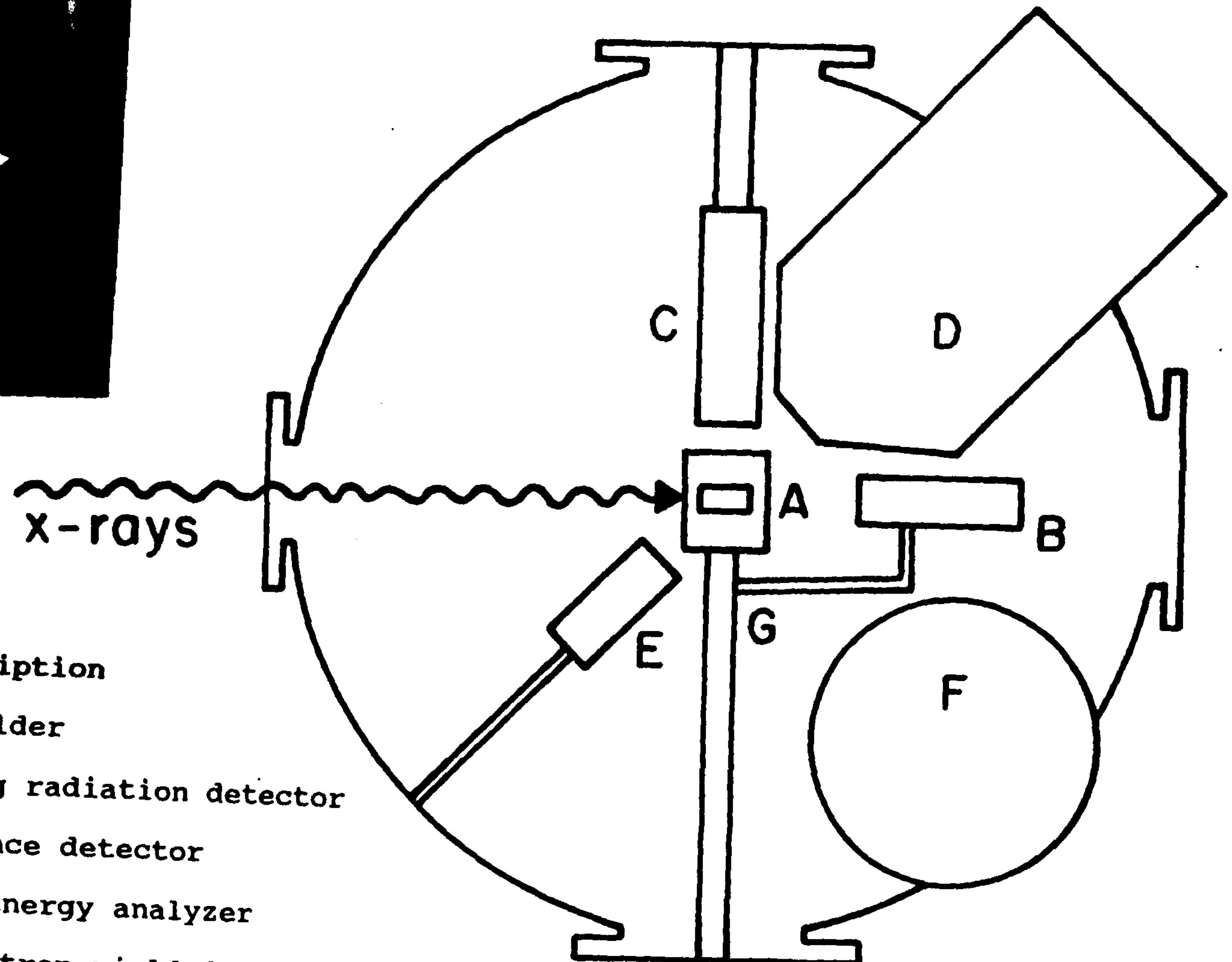
Although XAFS is a well established technique, experimenters must be familiar with some technical restrictions in order to extract unequivocal and useful information from the XAFS data, e.g. the problems associated with asymmetric pair distribution functions, anharmonic effects, choice of model compounds, and many-body effects. Misleading results have been reported by some researchers in the field. We emphasize that great care is required to make effective use of the XAFS spectroscopy. Our extensive experience in this area of research is useful in selecting the appropriate material systems which can realistically benefit from the XAFS studies.

All of our soft x-ray experiments were conducted with a high vacuum chamber as shown in Fig. 1. This setup allows measurements of x-ray fluorescence, electron yields, and scattering (both specular and diffuse reflection) at the same time as a function of photon energy or incidence angle. The soft x-ray fluorescence (e.g. from C, N, O, and F atoms) was measured with our new detector as shown in Fig. 2 [see Nucl. Instru. and Meth. A291 (1990) 135]. Hard x-ray measurements were made using a similar arrangement, except there was no need for a high vacuum chamber.

From the NEXAFS, or simply x-ray absorption spectroscopy (XAS) data, useful information on the local unoccupied electronic states pertaining to the x-ray absorbing atom in the solid can be obtained. By using different polarizations of the incident photons and single crystal samples, the symmetry property of the wave functions can be studied. This XAS method is element-specific and extremely valuable for probing the local distribution of charges on different oxygen and copper sites in the high-T_c superconductors.

We have found a special feature in the electronic structure on oxygen sites deduced





Chamber description

A - sample holder

B - scattering radiation detector

C - fluorescence detector

D - electron energy analyzer

E - total electron yield detector

F - cryostat

G - goniometer

Fig. 1

Detector description

1 - anode

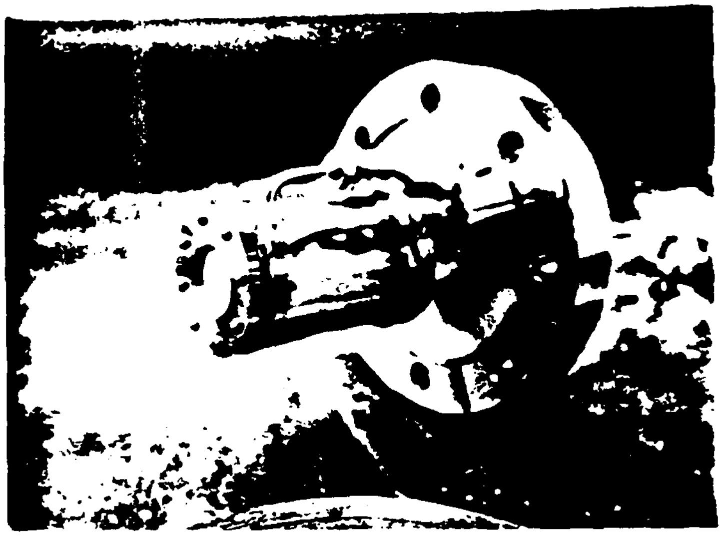
2 - cathode

3 - window

4 - o-ring

5 - body of detector

6 - electrical feedthrough



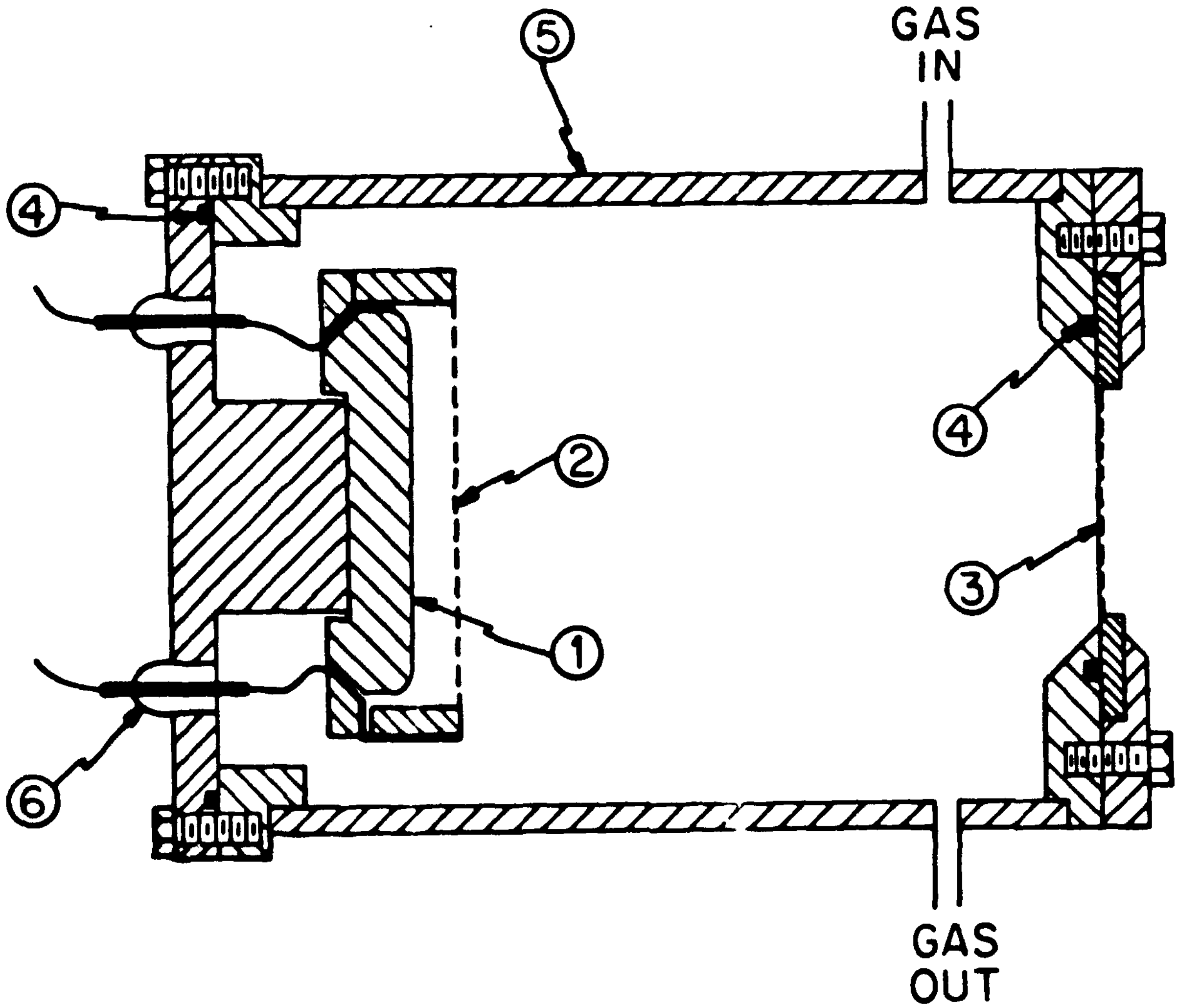


Fig. 2

from XAS measurements which is directly related to T_c. Peaks observed with incident photon energy in the region of 528-530 eV can be convincingly related to the structure of Hubbard bands in La-Sr-Cu-O and Tl-Ba-Ca-Cu-O systems. Also, by comparing the results of oxygen fluorescence yield (with a probing depth around 200 nm) and total electron yield (with a probing depth around a few nm), interesting information on oxygen deficiency near the superconductor surface can be obtained [details are presented in our publication Phys. Rev. B42 (1990) 2635]. We feel this technique is essential for studying the important connection between hole-doping and high-T_c superconductivity.

From EXAFS measurements, the interatomic distances, coordination numbers, and local disorder about a selected atomic species in complex systems can be studied. Since there is generally no long range structural ordering of the impurity atoms, the conventional diffraction techniques are not useful for probing the location of impurities. Due to the element selectivity and sensitivity to short-range-order structures, EXAFS is particularly useful for the investigation of chemically-doped superconductors. For example, we have applied the EXAFS technique to a study of Ag-doping in Y-Ba-Cu-O (YBCO) and our results provided the first convincing evidence that Ag atoms are indeed incorporated into the YBCO lattice [see J. Appl. Phys. 67 (1990) 353].

Our new soft x-ray detector offers a unique advantage for studying XAFS around the oxygen K edge. The oxygen K XAFS was not measured properly in the past because the experiments were mostly based on electron-yield detection method which is sensitive to surface conditions. Due to the presence of different stoichiometry and contamination on the surface, the electron-yield XAFS results are questionable even with samples prepared

by in-situ cleaving. In contrast, our fluorescence detector now allows a reliable measurement of *bulk* oxygen K XAFS which is insensitive to surface conditions. We view this as an important progress.

Grazing Incidence X-ray Reflectivity, Fluorescence, and Electron Yields

The angular dependence of x-ray reflectivity, fluorescence, and electron yields contains much useful information on surface and interface structures. This is particularly important for the study of layered materials, such as thin film heterojunctions, composite film-substrate structures, and superlattices. By measuring the reflectivity, fluorescence, and electron yields as a function of the incidence angle of incoming x-rays, the roughness and other microstructures (e.g. interdiffusion of constituent atoms, formation of clusters) on the sample surface as well as the interfaces can be probed nondestructively. Our group has been doing these experiments on a variety of composite-material systems and the previous results helped converting some of these techniques from basic studies to quantitative tools. A convenient data-analysis procedure has now been established that by a comparison with theoretical models taking into account the effects of x-ray scattering due to interfacial roughness, some appropriate parameters indicative of the interfacial roughness can be deduced. This problem of x-ray scattering from a multilayer material was studied earlier by Kiessig and Parratt but without considering the effects of interfacial roughness. By a modification of the Fresnel equations and incorporating a vector theory of scattering similar to the work by Vidal and Vincent, we have developed a method which can now be readily used for the determination of interfacial roughness in multilayer systems [see Phys. Rev. B38 (1988) 8579

and Phys. Rev. B42 (1990) 3829]. Our results obtained with several layered compounds have shown satisfactory agreement between theory and experiments. Preliminary results on the angular dependence of oxygen fluorescence yield from a high-T_c superconductor have been obtained; this is shown in Fig. 3 as an example. From this result we have estimated that oxygen deficiency of about 5% or more could occur near the surface of a YBCO film prepared by laser ablation.

(II-B). Studies of Transport and Magnetic Properties

Experimental investigations of transport and magnetic properties of chemically-doped high-T_c superconductors and films were made in our laboratories at Buffalo.

Transport Phenomena

Measurements of I-V curves and Hall effect as a function of magnetic field (H), temperature (T) and field orientation can reveal useful information on both the normal state properties and flux pinning.

Our results on transport studies of laser-ablated YBCO films with Ag, Fe, Co, Ni dopants indicate that the magnetic-field dependence of J_c can be divided into two different regimes from which the effects dominated by weak links and flux pinning can be studied separately. In the weak-link dominated regime, J_c is mainly a function of magnetic flux in the weak-link area, and it shows an averaged behavior of randomly distributed Josephson junctions. On the other hand, J_c in the flux-pinning dominated regime is mainly a function

of magnetic field which controls the free energy and force on flux lines [see Physica C169 (1990) 107]. This observation helps us to differentiate two important mechanisms in order to control the critical current density with different dopants added to the host material.

Magnetic Properties

Extensive measurements were made to investigate the variation of Tc, Jc, H_{c1}, H_{c2}, and magnetic relaxation effects by using a SQUID magnetometer (made by Quantum Design). Magnetic hysteresis curves and time dependence of magnetization M were measured as a function of H and T. Experiments were performed by first cooling the sample in zero magnetic field (ZFC) to the measurement temperature, the magnetic field was then applied and M was measured as a function of time t. Also, the same sample was cooled in a field (FC) to the measurement temperature, the field was removed and the relaxation of M was measured as a function of t. These steps are necessary in order to establish an experimentally-defined "critical state" to compare M(t) with theoretical predictions. Since the activation potential is in general a *nonlinear* function of field gradient and many different flux pinning processes might be present, M can be a complicated function of t, T, and H. Depending on the driving force on the flux, the activation potential derived from M(t) and resistivity measurements can be quite different. The problem can be further complicated by the possibility of flux entanglement and inhomogeneous distribution of pinning sites in the superconductor. A scaling behavior of pinning force was discovered which can be conveniently used to describe the effects of chemical doping on J_c.

III. PUBLICATIONS

The following papers based on our experimental results were published in refereed journals by the principal investigator (with his co-workers):

1. "Critical Current Density Enhancement and Moisture Destruction Studies of the Compound Superconductor Y-Ba-Cu-Ag-O" (with Y.D. Yao, A. Krol, C. Walters, S. Spagna, J. Althoff, S.C. Woronick, L.Y. Jang and F. Xu) Mat. Res. Soc. Symp. Proc. 99 (1988) 407.

2. "Short Range Order Structure Around Oxygen Atoms in the Superconductor Y-Ba-Cu-O" (with S.C. Woronick, W. Ng, A. Krol, B.X. Yang, R.L. Meng, P.H. Hor and C.W. Chu) Mat. Res. Soc. Symp. Proc. 99 (1988) 487.

3. "Local Environment Around Barium Atoms in the Superconductor Y-Ba-Cu-O" (with A. Krol, F. Xu, L.Y. Jang, C.J. Sher, S. Spagna and J. Althoff) Mat. Res. Soc. Symp. Proc. 99 (1988) 609.

4. "X-ray Absorption and Resistivity Studies of the Quinary-Compound Superconductor Y-Ba-Cu-O-S" (with S. Spagna, J. Althoff, Y.D. Yao, F. Xu, L.Y. Jang and A. Krol) Mat. Res. Soc. Symp. Proc. 99 (1988) 605.

5. "Determination of Oxygen Content in the High-Tc Superconductor Y-Ba-Cu-O" (with Z. Tao, D.E. Alburger, K.W. Jones, aand Y.D. Yao) Appl. Phys. Lett. 53 (1988) 1440.

6. "Short-Range-Order Structure in High-Tc Superconductors" Proc. First Asia-Pacific Conf. on Condensed Matter Physics (Singapore, 1988), p.136.

7. "Effects of Doping in the High-Tc Y-Based and Bi-Based Cuprates" (with Y.D. Yao) Proc. First Asia-Pacific Conf. on Condensed Matter Physics (Singapore, 1988), p. 248.

8. "Effects of Atomic Substitution in the High-Tc Superconductor Y-Ba-Cu-O System" (with Y.D. Yao, S. Spagna, and C. Walters) Proc. Int'l. Conf. Electronic Materials (Tokyo, 1988) p. 67.

9. "Effects of Chemical Doping and Stoichiometric Variation in High-Tc Superconductors" (with Y.D. Yao and L.W. Song) Int'l. J. Mod. Phys. B3 (1989) 573.

10. "Changes in Physical Properties of the High-Tc Superconductor Y-Ba-Cu-O Due to Cu Deficiency" (with Y.D. Yao, J.J. Simmins, R.L. Snyder, Z. Tao, and K.W. Jones) Mod. Phys. Lett. B3 (1989) 499.

11. "Oxygen K-Edge EXAFS of the High-Tc Superconductor Y-Ba-Cu-O" (with L.Y. Jang, W. Ng, A. Krol, S.C. Woronick, F. Xu, and Y.D. Yao) Physica B158 (1989) 488.

12. "Studies of Short Range Order Structure in the High-Tc Superconductor Y-Ba-Cu-O Using X-ray Absorption Techniques" (with A. Krol, L.Y. Jang, S.C. Woronick, F. Xu, and Y.D. Yao) Physica B158 (1989) 465.

13. "Probing the Effect of Atomic Substitution in the Superconductor Y-Ba-Cu-O by X-ray Absorption" (with L.Y. Jang, F. Xu, Y.D. Yao, A. Krol, and S.C. Woronick) Physica B158 (1989) 4.

14. "Probing the Short-Range-Order Structure in Solids Using Synchrotron Radiation" Proc. Third Asia Pacific Physics Conf. (Hong Kong, 1988), p. 408.

15. "Superconducting and Normal State Properties of BiSrCaCuO with Ag and Pb Doping" (with Y.D. Yao, Y.Y. Chen, J.H. Chen and C.L. Liu) Prog. in High Temp. Superconductivity 19 (1989) 249.

16. "Effects of Silver Doping in the High-Tc Superconductor System Y-Ba-Cu-O" (with Y.D. Yao, L.Y. Jang, F. Xu, A. Krol, L.W. Song, C.J. Sher, A. Darovsky, J.C. Phillips, J.J. Simmins, and R.L. Snyder) J. Appl. Phys. 67 (1990) 353.

17. "Magnetic Field Dependence of Critical Current Density in Silver-Doped Y-Ba-Cu-O Superconductors" (with L.W. Song) Proc. Third Annual Conf. on Superconductivity and Applications, Buffalo, (1989), p. 363.

18. "Studies of Microstructures in High-Tc Superconductors by X-ray Absorption Techniques" (with A. Krol, C.J. Sher, Z.H. Ming, C.S. Lin, L.W. Song, G.C. Smith, Y.Z. Zhu, and D.T. Shaw) Proc. Third Annual Conf. on Superconductivity and Applications, Buffalo, (1989), p. 281.

19. "A Low Pressure, Parallel Plate Avalanche Chamber for Detection of Soft X-ray Fluorescence" (with G.C. Smith and A. Krol) Nucl. Instr. and Meth. A291 (1990) 135.

20. "Oxygen Content Variation in Cation-Deficient and Chemically-Doped High-Tc Y-Ba-Cu-O and Bi-Sr-Ca-Cu-O Superconductors" (with L.W. Song, Y.D. Yao, Z. Tao, and K.W. Jones) Mat. Res. Soc. Symp. Proc. 169 (1990) 213.

21. "Soft X-ray Studies of Y-Ba-Cu-O Thin Films Prepared by Laser Ablation" (with A. Krol, G.C. Smith, C.J. Sher, D. Storch, L.W. Song, S. Witanachchi, Y.Z. Zhu, S. Patel, and D.T. Shaw) Mat. Res. Soc. Symp. Proc. 169 (1990) 505.

22. "Critical Current Density of Narrow Superconducting Thin Films Prepared by Laser Ablation Techniques" (with L.W. Song, Q.Y. Ying, J.P. Zheng, H.S. Kwok, Y.Z. Zhu, and D.T. Shaw) Mat. Res. Soc. Symp. Proc. 169 (1990) 89.

23. "X-ray Absorption Studies of Y-Ba-Cu-O and Bi-Sr-Ca-Cu-O Films at Oxygen K-Edge by Means of Fluorescence and Total-Electron-Yield: A Comparison of Two Techniques" (with A. Krol, C.S. Lin, Z.H. Ming, C.J. Sher, C.T. Chen, F. Sette, Y. Ma, G.C. Smith, Y.Z. Zhu, and D.T. Shaw) Phys. Rev. B42 (1990) 2635.

24. "X-ray Absorption Studies of Nd-Ce-Cu-O" (with A. Krol, C.S. Lin, Z.H. Ming, C.J. Sher, C.L. Lin, S.L. Qiu, J. Chen, J.M. Tranquada, M. Strongin, G.C. Smith, Y. Ma, F. Sette, C.T. Chen, Y.K. Tao, R.L. Meng, P.H. Hor, C.W. Chu, G. Gao, and J.E. Crow) Phys. Rev. B42 (1990) 4763.

25. "Superconducting Y-Ba-Cu-O Films on Metallic Substrates Using In-Situ Laser Deposition" (with E. Narumi, L.W. Song, F. Yang, S. Patel, and D.T. Shaw) Appl. Phys. Lett. 56 (1990) 2684.

26. "Critical Current Density of Ag-Doped Y-Ba-Cu-O Superconductors in Low Magnetic Fields" (with L.W. Song) Physica C169 (1990) 107.

27. "Oxygen 1s X-ray Absorption Spectroscopy of $Tl_2Ba_2Ca_2Cu_3O_{10}$ High-T_c Superconductors" (with A. Krol, C.S. Lin, Y.L. Soo, Z.H. Ming, Y. Ma, C.T. Chen, F. Sette, J.H. Wang, M. Qi, and G.C. Smith) Proc. Fourth Annual Conf. on Superconductivity and Applications, Buffalo, (1990), p. 206.

28. "Transport Measurements of $YBa_2(Cu_{0.98}M_{0.02})_3O_y$ (M=Fe, Co, Ni) Superconducting Thin Films" (with L.W. Song, E. Narumi, F. Yang, H.M. Shao, and D.T. Shaw), Proc. Fourth Annual Conf. on Superconductivity and Applications, Buffalo, (1990), p. 391.

29. "Critical Current Density Enhancement in $YBa_2Cu_3O_{6.8}$ Films on Buffered Metallic Substrates" (with E. Narumi, L.W. Song, F. Yang, S. Patel, and D.T. Shaw) Appl. Phys. Lett. 58 (1991) 1202.

30. "Transport Properties of $YBa_2(Cu_{0.98}M_{0.02})_3O_y$ (M=Fe, Co, Ni) Superconducting Thin Films" (with L.W. Song, E. Narumi, F. Yang, H.M. Shao, and D.T. Shaw), Physica C174 (1991) 303.

31. "Electronic States in La-Sr-Cu-O Probed by Soft-X-ray Absorption" (with C.T. Chen, F. Sette, Y. Ma, M.S. Hybertsen, E.B. Stechel, W.M. Foulkes, M. Schluter, S.W. Cheong, A.S. Cooper, L.W. Rupp, Jr., B. Batlogg, Y.L. Soo, Z.H. Ming, A. Krol) Phys. Rev. Lett. 66 (1991) 104.

32. "Short Range Order Structures in Y-Pr-Ba-Cu-O System Studied by X-ray Absorption Fine Structure (XAFS) Techniques" (with A. Krol, Z.H. Ming, C.S. Lin, Y.L. Soo, C.X. Gu, I.S. Yang, and C.C. Tsuei", Mat. Res. Soc. Symp. Proc. 209 (1991) 759.

33. "Microstructures in Y-Ba-Cu-O Thin Films Investigated by XAFS Techniques" (with A. Krol, Z.H. Ming, C.S. Lin, Y.L. Soo, C.X. Gu, E. Narumi, D.T. Shaw, and G.C. Smith), Mat. Res. Soc. Symp. Proc. 209 (1991) 873.

34. "Scaling Behavior of Flux-Pinning Force in Y-Ba-Cu-O Thin Films" (with L.W. Song, E. Narumi, M. Yang, F. Yang, D.T. Shaw), Physica C181 (1991) 239.

35. "Studies of Chemical Doping in High-Tc Superconductors", Proc. Miami Workshop on Electronic Structure and Mechanisms for High Temperature Superconductivity (Jan. 3-9, 1991). (Invited talk).